REMARKS

This amendment is respectfully submitted to place subject Application in condition for allowance. The Specification has been amended to correct typographical errors, and improve grammar and syntax. In particular, at page 5, line 13, the term "efficent" has been replaced with --efficient--. At page 5, line 30 the term "fractrions" has been replaced with -- fractions--. At page 26, line 22, the term "(37,248A)" has been replaced with --09/779,283--. At page 26, line 23, the term "(37,248B)" has been replaced with --09/779,286--. Claims 1, 9, 14, 15 and 19 have been amended to more distinctly claim the subject matter of the invention.

In particular, Claim 1 has been amended to recite that the mixture comprising the oxidation feedstock has a gravity ranging from about 10° API to about 100° API and is a product of a hydrotreating process for petroleum distillate, i.e., hydrotreated distillate. Support for this amendment is found in the Specification, for example, at page 15, lines 3 to 14.

In Claim 9 the term "high-boiling" has been deleted.

Claim 14 has been amended to recite the step of contacting all or at least a portion of the recovered organic phase with at least one solid sorbent comprising alumina.

Claim 15 has been amended to recite the step of contacting all or at least a portion of the recovered organic phase with at least one immiscible liquid comprising a solvent having a dielectric constant suitable to selectively extract oxidized sulfur-containing and/or nitrogen-containing organic compounds.

Claim 19 has been amended to recite the step of contacting all or at least a portion of the recovered organic phase with at least one immiscible liquid comprising an aqueous solution of a soluble basic chemical compound selected from the group consisting of

sodium, potassium, barium, calcium and magnesium in the form of hydroxide, carbonate or bicarbonate.

In view of the amendments submitted herein, it is the position of Applicants that the instant Application is in condition for allowance.

Claim Rejections - 35 U.S.C. § 112

In an Office Action mailed July 2, 2002, Claims 9 and 14 to 20 were rejected under 35 U.S.C. § 112, second paragraph. Applicants respectfully traverse these rejections.

In view of the amendments submitted above, it is the position of Applicants that instant Claims 9 and 14 to 20, inclusive, meet all requirements of 35 U.S.C. § 112.

Claim Rejections - 35 U.S.C. § 102

In the outstanding Office Action, Claims 1, 2, 4, 9 and 10 were rejected under 35 U.S.C. § 102(e), as being anticipated by U.S. Patent No 6,402,940 in the name of Alkis S. Rappas. Applicants respectfully traverse these rejections.

The Rappas reference of record describes a process for removing low amounts of organic sulfur from hydrocarbon fuels using an oxidizing/extracting solution of formic acid, a small amount of hydrogen peroxide, and no more than about 25 wt % water.

By contrast, instant Claim 1 recites: A process for the production of refinery transportation fuel or blending components for refinery transportation fuel, which process comprises:

providing an oxidation feedstock comprising a mixture of hydrocarbons, sulfur-containing and **nitrogen-containing organic compounds, which mixture has a gravity ranging from about 10° API to about 100° API and is a product of a hydrotreating process for petroleum distillate** (emphasis supplied);

contacting the oxidation feedstock with an immiscible phase comprising at least one organic peracid or precursors of organic peracid, in a liquid reaction mixture maintained substantially free of catalytic active metals and/or active metal-containing compounds and under conditions suitable for oxidation of one or more of the sulfur-containing and/or nitrogen-containing organic compounds; and

separating at least a portion of the immiscible peracid-containing phase from the reaction mixture; and

recovering a product comprising a mixture of organic compounds containing less sulfur **and/or less nitrogen** than the oxidation feedstock from the reaction mixture (emphasis supplied).

It is the position of Applicants that instant Claim 1 and dependent Claims 2, 4, 9 and 10, meet all requirements under 35 U.S.C. § 102(e).

In the outstanding Office Action, Claims 1-3, 9 and 10 were rejected under 35 U.S.C. § 102(b), as being anticipated by U.S. Patent No 1,972,102 in the name of William M. Malisoff. Applicants respectfully traverse these rejections.

The Malisoff reference of record describes a treatment for removal of sulfur from hydrocarbon oil by washing the oil with a treating solution containing acetic acid and hydrogen peroxide in water.

By contrast, instant Claim 1 recites: A process which comprises: providing an oxidation feedstock comprising a mixture of hydrocarbons, sulfur-containing and **nitrogen-containing**

organic compounds, which mixture has a gravity ranging from about 10° API to about 100° API and is a product of a hydrotreating process for petroleum distillate;. . . ; and

recovering a product comprising a mixture of organic compounds containing less sulfur **and/or less nitrogen** than the oxidation feedstock from the reaction mixture (emphasis supplied).

It is the position of Applicants that instant Claim 1 and dependent Claims 2, 3, 9 and 10, meet all requirements under 35 U.S.C. § 102(b).

Claim Rejections - 35 U.S.C. § 103

In the Office Action mailed July 2, 2002, Claim 4 was rejected under 35 U.S.C. § 103(a), as being unpatentable over U.S. Patent No. 1,972,102 in the name of William M. Malisoff, in view of U.S. Patent No 6,402,940 in the name of Alkis S. Rappas. Applicants respectfully traverse these rejections.

Additionally, in the Office Action mailed July 2, 2002, Claim 3 was rejected under 35 U.S.C. § 103(a), as being unpatentable over U.S. Patent No 6,402,940 in the name of Alkis S. Rappas in view of U.S. Patent No. 3,163,593 in the name of Alfred Bentley Webster, Norman James Herbert, and Richard Rigby (Webster et. al.). Applicants respectfully traverse these rejections.

Additionally, Claims 5 to 8 and 11 were rejected under 35 U.S.C. § 103(a), as being unpatentable over U.S. Patent No 6,402,940 in the name of Alkis S. Rappas in view U.S. Patent No 6,217,748 in the name of Shigeto Hatanaka, Osamu Sadakane, and Hideshi Iki and assigned to Nippon Mitsubishi Oil Corp. (JP), (Hatanaka et al.). Applicants respectfully traverse these rejections.

Additionally, Claims 12 to 20 were rejected under 35 U.S.C. § 103(a), as being unpatentable over U.S. Patent No 6,402,940 in the

name of Alkis S. Rappas in view U.S. Patent No 6,217,748 in the name of Shigeto Hatanaka, Osamu Sadakane, and Hideshi Iki and assigned to Nippon Mitsubishi Oil Corp. (JP), (Hatanaka et al.) as app0plied to claim 11 and further in view of Webster et. al. Applicants respectfully traverse these rejections.

An affidavit under 37 CFR § 1.131 of prior invention by Dr. William H. Gong on the 19th day of September 2002, identified as Paper No. 5, is hereby presented to further the prosecution of subject Application. In particular, the affidavit of Dr. Gong is a showing of facts which establish that reduction to practice of Applicants' invention, as described and claimed in subject application, was completed in the United States, prior to September 2000.

Applicants respectfully request Primary Examiner Griffin to withdraw all rejections under 35 U.S.C. § 103 (a) which rely upon the Rappas reference of record alone or in combination with the Malisoff, Webster et al., and/or Hatanaka et al. references of record.

An affidavit under 37 CFR § 1.131 of prior invention by Dr. George A. Huff Jr. on the 19th day of September 2002, identified as Paper No. 6, is hereby presented to further the prosecution of subject Application. In particular, the affidavit of Dr. Huff is a showing of facts which establish that reduction to practice of Applicants' invention, as described and claimed in subject application, was completed in the United States, prior to April 2000.

Applicants respectfully request Primary Examiner Griffin to withdraw rejections under 35 U.S.C. § 103 (a) which rely upon the Hatanaka et al. reference alone or in combination with the Rappas, and/or Webster et. al. references of record.

Base on the amendments submitted herein, Paper No. 5 and Paper No. 6, Applicants urge that Claims 1 to 20 inclusive, all claims now presented, are in condition for allowance. Applicant respectfully requests Primary Examiner Griffin to pass subject application for allowance.

Do not hesitate to contact Frederick S. Jerome whose telephone number is (630) 832-7974 (FAX (630) 832-7976) if additional assistance is needed regarding this paper or earlier papers for Applicants.

Applicants and their undersigned Attorney appreciate the attention and further consideration of this matter by Examiner Griffin.

Respectfully submitted,

Frederick S Jerome

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Version of Amended Specification with Markings to Show Changes Made

At page 5, kindly amend, lines 10 to 20, to read as follows:

Conventional hydrodesulfurization (HDS) catalysts can be used to remove a major portion of the sulfur from petroleum distillates for the blending of refinery transportation fuels, but they are not [efficent] efficient for removing sulfur from compounds where the sulfur atom is sterically hindered as in multi-ring aromatic sulfur compounds. This is especially true where the sulfur heteroatom is doubly hindered (e.g., 4,6-dimethyldibenzothiophene). Using conventional hydrodesulfurization catalysts at high temperatures would cause yield loss, faster catalyst coking, and product quality deterioration (e.g., color). Using high pressure requires a large capital outlay.

At page 5, kindly amend, lines 26 to 35, to read as follows:

The art is replete with processes said to remove sulfur from distillate feedstocks and products. One known method involves the oxidation of petroleum fractions containing at least a major amount of material boiling above a very high-boiling hydrocarbon materials (petroleum [fractrions] fractions containing at least a major amount of material boiling above about 550° F.) followed by treating the effluent containing the oxidized compounds at elevated temperatures to form hydrogen sulfide (500° F. to 1350° F.) and/or hydroprocessing to reduce the sulfur content of the hydrocarbon material. See, for example, U.S. Patent Number 3,847,798 in the name of Jin Sun Yoo and U.S. Patent Number 5,288,390 in the name of Vincent A. Durante. Such methods have proven to be of only limited utility since only a rather low degree of desulfurization is achieved. In addition, substantial loss of valuable products may result due to cracking and/or coke formation during the practice of these methods. Therefore, it would be advantageous to develop a

process which gives an increased degree of desulfuriztion while decreasing cracking or coke formation.

At page 26, kindly amend, lines 14 to 23, to read as follows:

Beneficially, all or a portion of the low-boiling fraction in substantially liquid form is diverted through conduit **32a** and into an optional oxygenation process unit **110** for catalytic oxidation in the liquid phase with a gaseous source of dioxygen, such as air or oxygen enriched air. For the purpose of the present invention, the term "oxygenation" is defined as any means by which one or more atoms of oxygen is added to a hydrocarbon molecule. Particularly suitable catalytic oxygenation processes are disclosed in commonly assigned U.S. Patent Application Serial Number 09/779,283 [(37,248A)] and U.S. Patent Application Serial Number 09/779,286 [(37,248B)].

Version of Amended Claims with Markings to Show Changes Made

1. (Once Amended) A process for the production of refinery transportation fuel or blending components for refinery transportation fuel, which process comprises:

providing <u>an</u> oxidation feedstock comprising a mixture of hydrocarbons, sulfur-containing and nitrogen-containing organic compounds, <u>which mixture has</u> a gravity ranging from about 10° API to about 100° API <u>and is a product of a hydrotreating process for petroleum distillate</u> [the mixture having];

contacting the oxidation feedstock with an immiscible phase comprising at least one organic peracid or precursors of organic peracid, in a liquid reaction mixture maintained substantially free of catalytic active metals and/or active metal-containing compounds and under conditions suitable for oxidation of one or more of the sulfur-containing and/or nitrogen-containing organic compounds; and

separating at least a portion of the immiscible peracid-containing phase from the reaction mixture; and

recovering a product comprising a mixture of organic compounds containing less sulfur and/or less nitrogen than the oxidation feedstock from the reaction mixture.

9. (Once Amended) The process according to claim 1 wherein the [high-boiling] oxidation feedstock consists essentially of material boiling between about 200° C. and about 425° C.

14. (Once Amended) The process according to claim 12 wherein the treating of recovered organic phase includes <u>contacting all or at least a portion of the recovered organic phase with</u> [use of] at least one solid sorbent comprising alumina.

15. (Once Amended) The process according to claim 12 wherein the treating of recovered organic phase includes <u>contacting all or at least a portion of the recovered organic phase with</u> [use of] at

least one immiscible liquid comprising a solvent having a dielectric constant suitable to selectively extract oxidized sulfur-containing and/or nitrogen-containing organic compounds.

19. (Once Amended) The process according to claim 12 wherein the treating of recovered organic phase includes contacting all or at least a portion of the recovered organic phase with [use of] at least one immiscible liquid comprising an aqueous solution of a soluble basic chemical compound selected from the group consisting of sodium, potassium, barium, calcium and magnesium in the form of hydroxide, carbonate or bicarbonate.

#4

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:	) Paper No. 6
APPLICANTS: WILLIAM A. GONG, MONICA R. REGALBUTO & GEORGE A. HUFF JR.	)
SERIAL NO: 09/779,285	) Group Art Unit: 1764
FILED: February 8, 2001	) Examiner: Walter D. Griffin
FOR: PREPARATION OF COMPONENTS FOR REFINERY BLENDING OF TRANSPORTATION FUELS	) Attorney Docket No.: 37,248

AFFIDAVIT UNDER 37 CFR § 1.131
of Dr. GEORGE A. HUFF Jr.

Assistant Commissioner for Patents
Washington, DC 20231

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231

on October 2 2002. By Carol M. Neth

(Date) **(Signature)**

CAROL M. NETH

(Typed or Printed Name of Person Signing Certificate)

1. I, GEORGEA. HUFF Jr., being duly sworn, depose and say:

2. That I reside at 823 Morven Court, Naperville, Illinois 60563.

3. In May 1984 I received a Bachelor of Science degree in Chemical Engineering from the University of Utah, Salt Lake, Utah, USA. I received a Doctor of Philosophy Chemical Engineering in 1982 from the Massachusetts Institute of Technology, Cambridge, Massachusetts, USA.

4. 1982 to 1984, I held the position of Assistant Professor of Chemical Engineering at M.I.T.

5. 1984 to 1986, I was a Research Engineer working in the Hydrotreating Group of the Catalysis Department for Shell Development at Westhollow Technical Center, Houston, Texas, USA.

6. From 1986 to the present, I have been employed by Amoco Chemical Company, now BP Amoco Chemical Company, a corporation of the State of Delaware, and have the position of Senior Research Associate. Among the professional honors which have been conferred on me are memberships in the North American Catalysis Society and the American Chemical Society.

7. I am one inventor of the claimed subject matter of the above identified patent application.

8. I have read U.S. Patent No 6,217,748 in the name of Hatanaka et al., and entitled PROCESS FOR HYDRODESULFURIZATION OF DIESEL GAS OIL.

9. Prior to April of 2000, our invention as described and claimed in the subject application was completed in the United States, as evidenced by the following Exhibits:

09/779,285 filed 2/8/01

a Pages 1 and 2 of memorandum titled PRODUCTION OF LOW SULFUR DIESEL (25 PPM AND 150 PPM) by S. G. McDaniel and M. A. Jandick for Amoco Petroleum Products, Naperville, Illinois, identified as EXHIBIT A, illustrates the key points of our selective hydrogenation of high-boiling hydrogenation feedstock whereby the incorporation of hydrogen into hydrocarbon compounds, sulfur-containing organic compounds, and/or nitrogen-containing organic compounds assists by hydrogenation removal of sulfur and/or nitrogen from components for refinery blending of transportation fuels.

b Table 1 : Properties of Feed, identified as EXHIBIT B.

c Table 2 : Properties of Catalyst, identified as EXHIBIT C.

d Table 4 : Properties for < 30 ppm Composite Sulfur Product, identified as EXHIBIT D.

10. As EXHIBIT A, illustrates, a hydrotreated desulfurized diesel having 375 ppm sulfur was used as the feed to the hydrotreating pilot plant to make products having reduced sulfur levels of about 150 ppm sulfur and less than 30 ppm sulfur. The feed was designated as LS-98. As stated on page 2, "The hydrotreated feed was difficult to desulfurize since 80 percent of the sulfur compounds boiled above 600° F. The majority of these sulfur compounds are dibenzothiophenes and substituted dibenzothiophenes. We had to run the unit with fresh catalyst at 680° F to achieve the 25 ppm product sulfur level."

11. Properties of the feed are summarized in Table 1, identified as EXHIBIT B.

12. The fresh catalyst used is one of the more active CoMo catalysts on the market for desulfurization of petroleum distillates. Selected properties of the catalyst are summarized in Table 2, identified as EXHIBIT C.

13. During the run, the majority of the sulfur samples were tested using analytical methods SPPM1640 and GCSBP2360. The

results were confirmed periodically by analyzing the same sample by method SXRF12740. Properties for <30 ppm Composite Sulfur Product are summarized in Table 4, identified as EXHIBIT D. These results demonstrate the effectiveness of our procedure in reducing the sulfur and nitrogen content of LS-98-150-A600, which originally contained 350 ? or 375 ppmw sulfur and 89 ppmw nitrogen.

15. Copies of the above referenced memorandum pages are attached as Exhibits. The Exhibits are a true copy, except that the dates thereof and unrelated subject matter have been blanked-out, but all the dates are prior to August 1999.

AND FURTHER AFFIANT SAYETH NOT.

George A. Huff Jr.

GEORGE A. HUFF Jr.

STATE OF ILLINOIS)
) SS.
COUNTY OF DUPAGE)

Sworn to and subscribed before me, a Notary Public, by said GEORGE A. HUFF Jr., on this 19th day of September 2002.

(SEAL)

Jane L. Cooper

Notary Public

"OFFICIAL SEAL"
JANE L. COOPER
Notary Public, State of Illinois
My Commission Expires 06/01/04

09/779,285 filed 2/8/01

EXHIBIT A GEORGE A. HUFF Jr. AFFIDAVIT UNDER RULE 1.131

MEMORANDUM

Amoco Petroleum Products
Naperville, Illinois 60566

S. G. McDaniel
M. A. Jandick

PRODUCTION OF LOW SULFUR DIESEL (25 PPM AND 150 PPM)

INTRODUCTION

testing. A desulfurized diesel from Whiting (LS-98) was used as the feed to the pilot plant to make seven barrels of 150 ppm sulfur diesel and seven barrels with less than 30 ppm sulfur. This memorandum documents feed and product analyses along with associated pilot plant processing conditions.

OPERATION

Feed

Hydrotreated HMD (LS-98 diesel) from the Whiting DDU was used as the feed to AU-125. Since the feed is already hydrotreated, the remaining 350 ppm sulfur is heavy: approximately 80% of the sulfur boils above 600°F. The properties for the feed as analyzed by the Amoco Research Center are listed in Table I.

Catalyst

We loaded 664 grams (950 cc) of fresh
catalyst and 150 cc silicon carbide into the reactor. This catalyst is currently used in the distillate desulfurization unit at the Amoco Yorktown refinery and is one of the more active CoMo catalysts on the market for desulfurization. The catalyst properties as tested at the Amoco Research Center are listed in Table II.

Conditions

EOR 740°F). The conditions for the pilot plant were as follows: pressure 500 to 550 psig, pure H_2, temperature 600°F-680°F. Sulfur samples were taken every day or two to monitor the quality of the product and detect upsets in the unit.

09/779,285 filed 2/8/01

EXHIBIT A GEORGE A. HUFF Jr. AFFIDAVIT UNDER RULE 1.131

PRODUCTS/RESULTS

Two product composites were produced for this project, a 150 ppm sulfur diesel and a maximum 30 ppm sulfur diesel. The actual sulfur levels were Composite #1--151 ppm and Composite #2--25 ppm, respectively. The product properties for both sulfur levels as analyzed at the Amoco Research Center in Naperville are listed in Tables III and IV, respectively. Barrels 2, 3, 4, 5, 7, 8, and 9 were blended to make Composite #1. Barrels 10, 11, 12, 13, 14, 16, 17, 18, and 19 were blended to make Composite #2. The other barrels were used to flush the blending tank before each operation.

The main parameter tracked during the run was product sulfur concentration in ppm. The majority of the sulfur samples were tested using analytical method SPPM1640. The results were confirmed periodically by sending the same sample in for SXRF1240. A plot of sulfur concentration per period is shown in Figure 1.

SUMMARY

The AU-125 pilot run was executed efficiently, and the composites produced were on target. The hydrotreated feed was difficult to desulfurize since 80% of the sulfur compounds boiled above 600°F. The majority of these compounds are dibenzothiophenes and substituted dibenzothiophenes. We had to run the unit with fresh catalyst at 680°F to achieve the 25 ppm product sulfur level. Most of the Amoco distillate desulfurization units (DDUs)

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SGM/MAJ/jmm/mkl/9872w

Attachments

Keywords: Hydrotreating, Diesel, Low Sulfur, Pilot Plant, DC-130

09/779,285 filed 2/8/01

EXHIBIT B GEORGE A. HUFF Jr. AFFIDAVIT UNDER RULE 1.131

TABLE I

PROPERTIES OF FEED (LS-98 DIESEL FROM WHITING)

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DI :T)	Sulfur by Boiling Pt. (GCSBP 2360)
Sulfur (ppm)	375	S PPM 1640	IBP 0.5%	270	440
Nitrogen (ppm)	89	N PPM 1560	1.0 %	292	453
Aromatic carbon (wt%)	16.5	NMRC 6831	5.0 %	355	522
API Gravity	34.66	FLAPIG 9710	10.0 %	384	554
Sp. gravity	0.8516	FLAPIG 9710	20.0 %	429	615
Cetane Index (4 point)	46.946	FILCETIND4/1	30.0 %	457	633
Carbon (wt%)	86.96	CHHIGH 1450	40.0 %	490	663
Hydrogen (wt%)	13.11	CHHIGH 1450	50.0 %	523	667
			60.0 %	549	676
			70.0 %	575	693
			80.0 %	605	705
			90.0 %	636	727
			95.0 %	663	746
			99.0 %	714	825
			FBP 99.5 %	733	855

09/779,285 filed 2/8/01

EXHIBIT C GEORGE A. HUFF Jr. AFFIDAVIT UNDER RULE 1.131

TABLE II
PROPERTIES OF CATALYST

			Metals Analysis for CHD-1682			
Physical Properties	Result	Test Code	Test Code: ICP 1340		Test Code: XRF 1040	
BET Surface Area m2/g	236	5170 SA	Na ppm		Na Wt%	-
Cum. Desorption cc/g	0.48	5170 SA	Mg ppm		Mg Wt%	-
Avg. Pore volume A	29	5170 SA	Al ppm	3[illegible]	Al Wt%	
Crush Strength #/mm	3.60	-	Si ppm		Si Wt%	
CBD g/cc	0.71	-	P ppm		P Wt%	
			K ppm	<	K Wt%	
			Ca ppm		Ca Wt%	
			Ti ppm		Ti Wt%	
			V ppm		V Wt%	
			Cr ppm		Mn Wt%	
			Mn ppm		Fe Wt%	
			Fe ppm		Co Wt%	
			Co ppm		Ni Wt%	
			Ni ppm		Zn Wt%	
			Zn ppm		Mo Wt%	
			Mo ppm		Sn Wt%	
			Pb ppm		Sb Wt%	
					Ba Wt%	
					La Wt%	
					Ce Wt%	
					Nd Wt%	

Property of Amoco Oil Company
Proprietary--To Be Maintained in Confidence

TABLE III

PROPERTIES OF 150 PPM COMPOSITE SULFUR PRODUCT

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DIST)	Sulfur by Boiling Pt. (GCSBP 2360)
Sulfur (ppm)	151	S PPM 1640	IBP 0.5 %	333	35
Nitrogen (ppm)	33	N PPM 1560	1.0 %	-	135
Aromatic carbon (wt%)	15.6	NMRC 6831	5.0 %	394	522
API Gravity	35	FLAPIG 9710	10.0 %	421	572
Sp. gravity	0.8498	FLAPIG 9710	20.0 %	448	645
Cetane Index (4 point)	46.836	FILCETIND4/1	30.0 %	473	663
Carbon (wt%)	86.45	CHHIGH 1450	40.0 %	494	668
Hydrogen (wt%)	13.07	CHHIGH 1450	50.0 %	512	676
			60.0 %	532	687
			70.0 %	552	695
			80.0 %	574	708
			90.0 %	603	733
			95.0 %	630	757
			99.0 %	-	851
			FBP 99.5 %	643	893

TABLE IV

PROPERTIES FOR < 30 PPM COMPOSITE SULFUR PRODUCT

Physical Properties	Result	Test Code	Volume Percent	IBP Distillation (FILD86DIST)	Sulfur by Boiling Point (GCSBP 2360)
Sulfur (ppm)	25	S PPM 1640	IBP 0.5 %	349	20
Nitrogen (ppm)	17	N PPM 1560	1.0 %	-	46
Aromatic carbon (wt%)	14.8	NMRC 6831	5.0 %	408	238
API Gravity	35.3	FLAPIG 9710	10.0 %	428	640
Sp. gravity	0.8485	FLAPIG 9710	20.0 %	453	661
Cetane Index (4 point)	47.813	FILCETIND4/1	30.0 %	474	668
Carbon (wt%)	86.72	CHHIGH 1450	40.0 %	495	673
Hydrogen (wt%)	13.12	CHHIGH 1450	50.0 %	514	688
			60.0 %	532	695
			70.0 %	552	706
			80.0 %	573	719
			90.0 %	600	741
			95.0 %	625	767
			99.0 %	-	857
			FBP 99.5 %	647	910

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:	) Paper No. 5
APPLICANTS: WILLIAM A. GONG, MONICA R. REGALBUTO & GEORGE A. HUFF JR.	)
SERIAL NO: 09/779,285	) Group Art Unit: 1764
FILED: February 8, 2001	) Examiner: Walter D. Griffin
FOR: PREPARATION OF COMPONENTS FOR REFINERY BLENDING OF TRANSPORTATION FUELS	) Attorney Docket No.: 37,248

AFFIDAVIT UNDER 37 CFR § 1.131
of Dr. WILLIAM H. GONG

Assistant Commissioner for Patents
Washington, DC 20231

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 20231

on October 2 2002. By Carol M. Neth

(Date) **(Signature)**

CAROL M. NETH

(Typed or Printed Name f Person Signing Certificate)

09/779285 filed 2/8/01

1. I, WILLIAM H. GONG, being duly sworn, depose and say:

2. That I reside at 900 Fairfield Avenue, Elmhurst, Illinois 60126.

3. In May 1984 I received a Bachelor of Science degree in Chemistry from Illinois State University at Normal, Illinois, USA. I received a Doctor of Philosophy degree in Organic Chemistry in 1990 from the Iowa State University at Ames, Iowa, USA

4. From June 11, 1990 to the present, I have been employed by Amoco Chemical Company, now BP Amoco Chemical Company, a corporation of the State of Delaware, and have the position of Research Associate.

5. I have worked in the development of new catalytic oxidation process for distillate fuel streams as well as alkylaromatic feedstocks.

6. I am one inventor of the claimed subject matter of the above identified patent application.

7. I have read U.S. Patent No 6,402,940 in the name of Alkis S. Rappas, and entitled PROCESS FOR REMOVING LOW AMOUNTS OF ORGANIC SULFUR FROM HYDROCARBONFUELS.

8. Prior to September 2000, our invention as described and claimed in the subject application was completed in the United States, as evidenced by the following Exhibits:

a Laboratory Notebook 20164, pages 37 and 38, identified as EXHIBIT A, illustrates the key points of my oxidation and simultaneous extraction of a high boiling fraction of diesel fuel.

b Laboratory Notebook 20453, pages 44 to 47, identified as EXHIBIT B, illustrates the key points of my liquid-liquid

extraction of an oxidized high boiling fraction of diesel fuel to remove residual sulfur.

9. Under my supervision, Mrs. Christine Peaches, a BP Amoco Research Technician, conducted an oxidation-simultaneous extraction experiment described in an Amoco Research Notebook 20164, pages 37 to 38. In this experiment, Mrs. Peaches created a biphasic mixture comprising of 100 g of a high boiling fraction of diesel fuel (a.k.a. LS-98-25-A600 but also labeled as "LS-98-150-A600" which fraction boiled above 600° F (316° C) and contained 580 ppmw sulfur), 34 milliliter of 30 percent by weight hydrogen peroxide, 50 milliliter of glacial acetic acid (HOAc), and 50 milliliter of distilled and deionized water (D&D water). This mixture was created in a 3-neck, round bottom flask equipped with an overhead agitator, a water-cooled reflux condenser, a nitrogen inlet and outlet, and a heating mantel.

10. The mixture was heated to boiling (approximately 210° F) with an agitation rate fixed at 200 rpm, and was sustained at this temperature for 120 minutes (2 hours). After this heating period had elapsed, the heating mantel was switched off and the agitation was temporarily ceased to permit a disengagement of the diesel layer from the aqueous acetic acid layer. The top layer, the oxidized and extracted diesel immiscible layer, was sampled for sulfur analysis. After the sampling, the agitation was resumed until the mixture was cooled. Approximately 0.1 grams of manganese dioxide was added to the mixture to decompose any unused peroxide in the aqueous acetic acid layer. The entire reactor content was transferred to a storage container. The bottom, aqueous acetic acid layer, was sampled for sulfur analysis.

11. The oxidized and extracted diesel layer was determined to contain 355 ppmw sulfur (LIMS sample number 152051) while the bottom, aqueous acetic acid layer was determined to contain 41 ppmw sulfur (LIMS sample number 152084). These results demonstrate a desulfurization of the diesel layer due to oxidation

and simultaneous extraction of sulfur into the aqueous acetic acid layer.

12. As EXHIBIT B, illustrates, Mr. Donald J. Maciejewski, a BP Amoco Research Technician, conducted a liquid-liquid extraction of a hydrogen peroxide, acetic acid-based oxidation of a diesel fuel which boiled above 600 OF (316° C) and contained 580 ppmw sulfur and 147 ppmw nitrogen. The experiment required the preparation of a large volume (approximately one liter) of an "oxidized and simultaneously extracted oxidized diesel fuel" (from this point on known as the "oxidized diesel") to test the concept of the liquid-liquid extraction. This oxidized diesel fuel was prepared according to the instructions documented in Research Notebook number 20453, pages 44 to 46. The oxidized diesel fuel product obtained from this procedure was significantly desulfurized and denitrogenated as the product fuel contained 143 ppmw sulfur and 4 ppmw nitrogen (LIMS sample manager number 155378). The aqueous acetic acid layer separated after the oxidation-extraction procedure was found to have a sulfur concentration of 252 ppmw or 0.0252 percent by weight (LIMS sample manager number 155377), thus demonstrating that during the oxidation, an effective extraction of the oxidized diesel impurities occurred. After the oxidized diesel fuel was dehydrated by treatment with molecular sieves, the oxidized product is then identified as the "dried" diesel product.

13. The "dried" diesel product was then submitted for solubility testing in four different liquid-liquid solvent candidates. This work by Mr. Donald J. Maciejewski, is described in Research Notebook number 20453 on page 47. The test was to result in the selection of a feasible solvent which must be immiscible with the diesel product and that after mixing, the diesel product must separate quickly from the solvent layer.

14. Mr. Maciejewski found that methanol met the two criteria and his observations are recorded in Amoco Research

Notebook number 20453 on page 47. Subsequent work by Mr. Maciejewski demonstrated that after one extraction with methanol, the "dried" oxidized, methanol extracted product contain only 35 ppmw sulfur and 3 ppmw nitrogen (LIMS sample manager number 157666). These results demonstrate the effectiveness of my procedure in reducing the sulfur and nitrogen content of LS-98-150-A600, which originally contained 580 ppmw sulfur and 147 ppmw nitrogen.

15. Copies of the above referenced Laboratory Notebook pages are attached as Exhibits. The Exhibits are a true copy, except that the dates thereof and unrelated subject matter have been blanked-out, but all the dates are prior to September 2000.

AND FURTHER AFFIANT SAYETH NOT.

William H. Gong

WILLIAM H. GONG

STATE OF ILLINOIS)
) SS.
COUNTY OF DUPAGE)

Sworn to and subscribed before me, a Notary Public, by said WILLIAM H. GONG, on this 19th day of September 2002.

Jane L. Cooper

Notary Public

(SEAL)

"OFFICIAL SEAL"
JANE L. COOPER
Notary Public, State of Illinois
My Commission Expires 06/01/04

09/779285 filed 2/8/01

EXHIBIT A WILLIAM H. GONG, AFFIDAVIT UNDER RULE 1.131

20164

Project No. R00012-4:01 Date of Work ______ Continued From Page ______

Title or Purpose: Hydrogen Peroxide Oxidation of High Boiling Fraction

KEYWORDS: Diesel Fuel (LS-98-25-A600)

Run #20164-037-1

Flask Charged:

LS-98-150-A600	—	100g.
30% H_2O_2 (ML)	—	340
Glacial HOAC (ML)	—	50
D+D Water (ML)	—	50

Into a 250ML, three-neck round bottom flask equipped with a reflux condenser, mechanical agitator, a nirogen inlet and outlet set at 1.0 cc are charged the above, and heated at 100°C for 2hrs, stirred also at 20rpm. After the end of the 2hrs are up, from the top layer is sampled with 15ML and stored into a plastic 4-dram vial with the cap lossely fixed in case there are residual peroxides decomposing. Sample are transfered to T.J. Barnes for submit to Sample manager. The sample stirred and cooled to room temperature. Once at room temperature used 0.1g of manganese dioxide is added to decompose of the excess hydrogen peroxide and than stirred for 10 mintues more. Entire sample is then poured into a bottle with a vented cap.

(OVER)

Work By Chris Peaches Continued on Page 38

Recorded By Chris Peaches Date ______

Project No. R100012-4:01 Date of Work____ Continued From Page____

Title or Purpose:

KEYWORDS:

Cleanup Equiment:
The (Flask, Agitator, thermometer, Condenser) Are then rinsed with acetone and disposed According to Safety Proedure. After the organic residue has been removed, the equipment is than rinsed with distilled WATER and allowed to air dry.

Sample # 20164-037-1
WEIGHT For 15ML Sample
11.939g. 12.447g.
FINAL LARge Bottle Sample WT: 210.024g.

Work By Chris Peaches Continued on Page

Recorded By Chris Peaches Date

Project No.________ Date of Work_ ___ Continued From Page________

Title or Purpose: OXIDATIVE DESULFURIZATION OF LS-98-150-A600 (600 TO FBP

KEYWORDS:

Oxidative Desulfurization of LS-98-150-A600 (600 to FBP)

Note: This procedure is for a one-liter scale and can be scaled proportionally to equipment of different sizes.

Procedure

Into a three (3) or four (4) liter, three neck, round bottom flask equipped with a water-jacketed reflux condenser, a mechanical stirrer, a nitrogen inlet and outlet, a temperature controller (eurotherm and thermocouple) and a heating mantel, are charged with:

- one (1) kg of LS-98-150-A600 >600+
- one (1) liter of glacial acetic acid
- 170 mL of 30% hydrogen peroxide (H_2O_2).

A slight flow of nitrogen is initiated and this gas then slowly sweeps over the surface of the reactor content. The agitator is started to provide efficient mixing and the contents are heated. Once the temperature reaches 200 °F, the contents are heated for 120 minutes at this temperature.

After the reaction time has elapsed, the contents are continuing to be stirred with the heating mantel turned off and removed. At approximately 170 °F, the agitator is stopped momentarily while approximately 1 g of manganese dioxide (MnO_2) is added through one of the necks of the round bottom flask to the biphasic mixture to decompose any unreacted hydrogen peroxide. Mixing of the contents with the agitator is then resumed until the temperature of the mixture has cooled to approximately 120 °F. The agitation is ceased to allow both organic (top) and aqueous (bottom) layers to separate (should occur immediately).

The bottom layer is removed and isolated either by vacuum with the aid of a dip tube or some other equivalent procedure. Note: It isn't necessary to remove every last bit of the bottom layer; leaving behind a bit of this is not detrimental to the following procedure. The bottom layer is to be saved for further analysis. It is necessary that the aqueous acetic acid layer is stored in a lightly capped bottom to permit the evolution of oxygen.

Into the reactor is charged with 500 mL of saturated aqueous sodium bicarbonate to neutralize the organic layer (**CAUTION**: WHEN ADDING THIS SOLUTION, BE CERTAIN THAT THE AGITATOR IS STIRRING SLOWLY AND ADD THIS SOLUTION SLOWLY AS GAS (CO_2) EVOLUTION WILL OCCUR INITIALLY). After the bicarbonate solution has been added, allow the mixture to stir rapidly for five to ten minutes to neutralize any remaining acetic acid. Remove the bottom, aqueous layer, and isolate the top layer. Dry the organic material over anhydrous 3 angstrom

Work By AJ Mucyeski ________ Continued on Page 45

Recorded By ________

Read and Understood By ________

09/779285 filed 2/8/01

EXHIBIT B WILLIAM H. GONG, AFFIDAVIT UNDER RULE 1.131

20453

Project No.________ Date of Work________ Continued From Page 44

Title or Purpose: OXIDATIVE DESULFURIZATION OF LS-98-150 A600 (600 TO FBP)

KEYWORDS:

molecular sieve. After the material has been dehydrated by the molecular sieve, remove the sieve by filtration and isolate the filtrate.

Analytical Chemistry

1. Submit a sample of the first aqueous acetic acid layer for OELECOSHT.
2. Submit a sample of the organic layer for the following: OELECOSHT, OENPPMWOIL, CCTAN

SODIUM BICARBONATE AQUEOUS SATURATED SOLUTION
90.01 g

LS-98-150 FRACTION 5 POT
1160 ml = 1000.9 g
1 L GLACIAL ACETIC ACID 170 ml 30% HYDROGEN PEROX.
9:50 REACTOR = 204°F (OFF AT 11:50 AM)
12:15 PRODUCT AT 175°F ADDED 1.0049 g MANGANESE (IV) OXIDE 99.99.
TURN MIXER ON AND LET SOLUTION COOL TO 120°F
14:40 REACTOR AT 115°F USED DIP TUBE TO REMOVE BOTTOM LAYER 20453-45-1 (AQUEOUS ACETIC ACID BTM LAYER)

ADDED 500 ml OF SATURATED SODIUM BICARBONATE SOLUTION SLOWLY WHILE MIXTURE WAS TURNING AT 60 RPM.
AFTER ALL THE SODIUM BICARBINATE WAS ADDED THE STIRRER WAS INCREASED TO 200 RPM FOR 10 MINUTES.
MIXTURE WAS POURED INTO A 2000 ML SEPARATORY FUNNEL TO ALLOW THE LAYERS TO SEPARATE.
20453-45-2 AQUEOUS SODIUM BICARBONATE
20453-45-3 ORGANIC LAYER

Work By ________ Continued on Page 46

Recorded By DJ Macgregor Date ________

Read and Understood By Date

Project No.____ Date of Work____ Continued From Page 45

Title or Purpose:____

KEYWORDS:

DRIED THE ORGANIC LAYER BY ADDING 3A MOLECULAR SIEVE TO THE BOTTLE AND LET SIT OVERNIGHT

FILTERED THE ORGANIC LAYER THROUGH MILLIPORE LC 10.0μm FILTER

20453-046-1

1712.9	1684.3
840.3 g	840.3
872.6 g	844.0 g

ANALYTICAL

ACETIC ACID 20453-045-1

OELECOSHT (SULFUR, LECO, HIGH TEMP, D4239) 10 ml

JOB ID NAP20002OJM06-07227

JOB DESCR. DIESEL FUEL FROM B. GONG

ID NUMERIC

155377 20453-045-1

ORGANIC LAYER (TEMPLATE GONG DIESEL)

~~OEL~~ OELECOSHT SULFUR, LECO HIGH TEMP, D4239 10 ml

OENPPMWOIL NITROGEN PPM-W IN OILS & WATER 2g

CC7AN TOTAL ACID # 20g

NAP20002OJM06-07228

ID NUMERIC

155378 20453-046-1

Work By ____ Continued on Page ____

Recorded By ____ Date ____

Read and Understood By ____ Date ____

Project No. RI-00012-4101 Date of Work_ Continued From Page_

Title or Purpose: METHANOL EXTRACTIONS

KEYWORDS: SOLUBILTY, EMULSION, DIESEL FUEL

PURPOSE: TO TEST SOLUBILITY OF "DRIED" DIESEL FROM PEROXIDE OXIDATION WITH METHANOL

TEST 1 1:1 VOL RATIO OF THE DRIED DIESEL FROM THE PEROXIDE OXIDATION AND METHANOL

EMULSION BREAKS FAIRLY QUICK ≈ 3 MINUTES

TEST 2 MAKE UP A SOLUTION OF 70% METHANOL/30% WATER (BY VOLUME)

MAKE UP 1:1 VOL RATIO OF DRIED DIESEL FROM PEROXIDE OXIDATION WITH ABOVE 70% MeOH/30% WATER

SLOW TO SEPARATE (≈ 20 MINUTES)

TEST 3 SATURATE 70% METHANOL/30% WATER WITH NaCl

MAKE UP 1:1 VOL RATIO OF DRIED DIESEL FROM PEROXIDE OXIDATION WITH ABOVE NaCl SOLUTION

NOTE: FILTER SATURATED NaCl SOLUTION THROUGH A MILLIPORE, LC, 10μm PORE SIZE, BEFORE MIXING WITH THE DIESEL FUEL

GOOD EMULSION BREAK, QUICK

TEST 4 1:1 VOL RATIO DRIED DIESEL FROM PEROXIDE OXIDATION WITH WATER

Work By _ Continued on Page _

Recorded By _ Date _

Read and Understood By _ Date